

PCT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
United States Patent and Trademark
Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

| | |
|---|---|
| Date of mailing (day/month/year) 06 July 2000 (06.07.00) | |
| International application No. PCT/GB99/03580 | Applicant's or agent's file reference P/23259.WO/ICB |
| International filing date (day/month/year) 29 October 1999 (29.10.99) | Priority date (day/month/year) 30 October 1998 (30.10.98) |
| Applicant ASSOUMANI, Mohamed, Bakri | |

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:
26 May 2000 (26.05.00)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

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|--|--|
| <p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No.: (41-22) 740.14.35</p> | <p>Authorized officer S. Mafla</p> <p>Telephone No.: (41-22) 338.83.38</p> |
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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

REC'D 16 FEB 2001

WIPO PCT

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|--|--|---|--|
| Applicant's or agent's file reference 23259.WO/ICB | | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) | |
| International application No. PCT/GB99/03580 | International filing date (day/month/year) 29/10/1999 | Priority date (day/month/year) 30/10/1998 | |
| International Patent Classification (IPC) or national classification and IPC A23L1/00 | | | |
| Applicant AQUACAL LIMITED et al. | | | |

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.


2. This REPORT consists of a total of 5 sheets, including this cover sheet.

☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☒ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☒ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☒ Certain observations on the international application

| | |
|---|--|
| Date of submission of the demand 26/05/2000 | Date of completion of this report 13.02.2001 |
| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | Authorized officer Vernier, F Telephone No. +49 89 2399 8646 |



1. The present communication is an Annex to the invitation to pay additional fees (Form PCT/ISA/206). It shows the results of the international search established on the parts of the international application which relate to the invention first mentioned in claims Nos. 1-8
2. This communication is not the international search report which will be established according to Article 18 and Rule 43.
3. If the applicant does not pay any additional search fees, the information appearing in this communication will be considered as the result of the international search and will be included as such in the international search report.
4. If the applicant pays additional fees, the international search report will contain both the information appearing in this communication and the results of the international search on other parts of the international application for which such fees will have been paid.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|------------|--|-----------------------|
| A | WO 98 33508 A (AQUACAL LIMITED) 6 August 1998 (1998-08-06) cited in the application --- | 1 |
| A | FR 2 201 040 A (S.A.R.A.P. - C.E.D.I.A.) 26 April 1974 (1974-04-26) cited in the application --- | |
| A | DE 29 47 186 A (BIOLABOR WALTER BRACHMANN)) 27 May 1981 (1981-05-27) claims 1,2 --- | 1 |
| A | BE 693 094 A (BOUCHLET A.) 3 July 1967 (1967-07-03) page 4, line 29-31 page 5, line 23-27 page 6, line 8,9; claims 1,3,5 --- | 1-8 |
| A | DATABASE WPI Section Ch, Week 199314 Derwent Publications Ltd., London, GB; Class D13, AN 1993-111855 XP002132742 & JP 05 049446 A (KANKYO HOZEN KENKYUSHO YG), 2 March 1993 (1993-03-02) abstract ----- | 1 |

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *&* document member of the same patent family

Patent Family Annex

Information on patent family members

International Application No

T/GB 99/03580

| Patent document cited in search report | | Publication date | Patent family member(s) | Publication date |
|---|---|---------------------|---|--|
| WO 9833508 | A | 06-08-1998 | AU 5676498 A EP 0966295 A ZA 9800881 A | 25-08-1998 29-12-1999 03-08-1998 |
| FR 2201040 | A | 26-04-1974 | NONE | |
| DE 2947186 | A | 27-05-1981 | NONE | |
| BE 693094 | A | 03-07-1967 | FR 5576 M CH 500711 A DE 1617340 A GB 1113318 A IT 954012 B LU 52903 A NL 6702168 A | 02-01-1968 31-12-1970 25-03-1971 30-08-1973 30-03-1967 15-08-1967 |
| JP 5049446 | A | 02-03-1993 | NONE | |

INVITATION TO PAY ADDITIONAL FEES

International application No.

PCT/GB 99/03580

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1-8

Use of algae for foodstuff

2. Claim : 9

cosmetic material containing algae

The Application contains a priori two different problems namely: the provision of improved foodstuff containing calcium and the provision of a cosmetic material.

The common essential feature linking both inventions is the use of the corallinaceae algae; since this common feature is already known from the prior art, see current application page 1, lines 16-19 and due to the fact that no other technical feature can be regarded as special technical feature in the sense of rule 13.2 PCT, the ISA is of the opinion that there is no single inventive concept underlying the plurality of claimed inventions of the present application in the sense of Rule 13.1 PCT.

Consequently there is a lack of unity and the different inventions, not belonging to a common inventive concept, are formulated as the different subjects as the communication pursuant to Article 17(3)(a) PCT.

The search has been carried out only with respect to the first subject. Searching the invention of the second group would have caused major additional searching efforts.

WRITTEN OPINION

International application No. PCT/GB99/03580

I. Basis of the opinion

1. This opinion has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".*):

Description, pages:

1-28 as originally filed

Claims, No.:

1-9 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

III. Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non-obvious), or to be industrially applicable have not been and will not be examined in respect of:

- ☐ the entire international application,
☒ claims Nos. 9,

because:

- ☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international preliminary examination (*specify*):

☐ the description, claims or drawings (*indicate particular elements below*) or said claims Nos. are so unclear that no meaningful opinion could be formed (*specify*):

WRITTEN OPINION

International application No. PCT/GB99/03580

- ☐ the claims, or said claims Nos. are so inadequately supported by the description that no meaningful opinion could be formed.
- ☒ no international search report has been established for the said claims Nos. 9.

IV. Lack of unity of invention

1. In response to the invitation (Form PCT/IPEA/405) to restrict or pay additional fees, the applicant has:
- ☐ restricted the claims.
- ☐ paid additional fees.
- ☐ paid additional fees under protest.
- ☒ neither restricted nor paid additional fees.
2. ☐ This Authority found that the requirement of unity of invention is not complied with for the following reasons and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees:
3. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this opinion:
- ☐ all parts.
- ☒ the parts relating to claims Nos. 1-8.

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | |
|-------------------------------|------------|
| Novelty (N) | Claims 1-8 |
| Inventive step (IS) | Claims 1-8 |
| Industrial applicability (IA) | Claims |

2. Citations and explanations

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Item IV

1. With respect to lack of unity of invention, the examiner agrees with the opinion expressed with the international search report. Only claims 1-8 (use of algae for foodstuff) will therefore be the object of a substantive examination (Rule 13 PCT).

Item V

2. The subject-matter of present claims 1-8 (use) is not novel (Article 33(2) PCT) since document D1=BE 693 094 A discloses the use of calcareous residues of algae as an oligoelement (calcium) supplement, to improve the organoleptic properties of foods and as a stabiliser in flours (see in particular: page 5, line 19 - page 6, line 9 and claims 1-2).
3. The subject-matter of present claims 1-8 (use) meets the requirement of Article 33(4) PCT, since it can be applicable in the food industry.

Item VII

4. To meet the requirements of Rule 5.1(a)(ii) PCT, the document D1 should have been identified in the description and the relevant background art disclosed therein should have been briefly discussed.

Item VIII

5. The passages of the description related to cosmetic products do not fall within the scope of the claims. This inconsistency between the claims and the description leads to doubt concerning the matter for which protection is sought, thereby rendering the claims unclear (Article 6 PCT).
6. The term "AquaMin" used in the present description appears to be a registered trade mark and should be identified as such (Article 6 PCT).



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Europäisches
Patentamt

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Patent Office

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Correspondence with the EPO on PCT Chapter II demands

In order to ensure that your PCT Chapter II demand is dealt with as promptly as possible you are requested to use the enclosed self-adhesive labels with any correspondence relating to the demand sent to the Munich Office.

One of these labels should be affixed to a prominent place in the upper part of the letter or form etc. which you are filing.

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/03580

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).):*

Description, pages:

1-28 as originally filed

Claims, No.:

1-9 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/03580

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

III. Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

1. The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non-obvious), or to be industrially applicable have not been examined in respect of:

☐ the entire international application.

☒ claims Nos. 9.

because:

☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international preliminary examination (*specify*):

☐ the description, claims or drawings (*indicate particular elements below*) or said claims Nos. are so unclear that no meaningful opinion could be formed (*specify*):

☐ the claims, or said claims Nos. are so inadequately supported by the description that no meaningful opinion could be formed.

☒ no international search report has been established for the said claims Nos. 9.

2. A meaningful international preliminary examination report cannot be carried out due to the failure of the nucleotide and/or amino acid sequence listing to comply with the standard provided for in Annex C of the Administrative Instructions:

☐ the written form has not been furnished or does not comply with the standard.

☐ the computer readable form has not been furnished or does not comply with the standard.

IV. Lack of unity of invention

1. In response to the invitation to restrict or pay additional fees the applicant has:

☐ restricted the claims.

☐ paid additional fees.

☐ paid additional fees under protest.

☒ neither restricted nor paid additional fees.

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/03580

2. ☐ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.
3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is
- ☐ complied with.
- ☒ not complied with for the following reasons:
see separate sheet
4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:
- ☐ all parts.
- ☒ the parts relating to claims Nos. 1-8.

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | |
|-------------------------------|-----------------|
| Novelty (N) | Yes: Claims |
| | No: Claims 1-8 |
| Inventive step (IS) | Yes: Claims |
| | No: Claims 1-8 |
| Industrial applicability (IA) | Yes: Claims 1-8 |
| | No: Claims |

2. Citations and explanations
see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/03580

2. ☐ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.
3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is
- ☐ complied with.
- ☒ not complied with for the following reasons:
see separate sheet
4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:
- ☐ all parts.
- ☒ the parts relating to claims Nos. 1-8.

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | |
|-------------------------------|-----------------|
| Novelty (N) | Yes: Claims |
| | No: Claims 1-8 |
| Inventive step (IS) | Yes: Claims |
| | No: Claims 1-8 |
| Industrial applicability (IA) | Yes: Claims 1-8 |
| | No: Claims |

2. Citations and explanations
see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/03580

Item IV

1. With respect to lack of unity of invention, the examiner agrees with the opinion expressed with the international search report. Only claims 1-8 (use of algae for foodstuff) were therefore the object of a substantive examination (Rule 13 PCT).

Item V

2. The subject-matter of present claims 1-8 (use) is not novel (Article 33(2) PCT) since document D1=BE 693 094 A discloses the use of calcareous residues of algae as an oligoelement (calcium) supplement, to improve the organoleptic properties of foods and as a stabiliser in flours (see in particular: page 5, line 19 - page 6, line 9 and claims 1-2).
3. The subject-matter of present claims 1-8 (use) meets the requirement of Article 33(4) PCT, since it can be applicable in the food industry.

Item VII

4. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the document D1 has not been identified in the description and the relevant background art disclosed therein has not been briefly discussed.

Item VIII

5. The passages of the description related to cosmetic products do not fall within the scope of the claims. This inconsistency between the claims and the description leads to doubt concerning the matter for which protection is sought, thereby rendering the claims unclear (Article 6 PCT).
6. The term "AquaMin" used in the present description appears to be a registered trade mark and should have been identified as such (Article 6 PCT).

18 SEP 2000

From the:
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

IAIN C. BAILLIE
LANGNER PARRY
52-54 High Holborn
London WC1V 6RR
GRANDE BRETAGNE

PCT

WRITTEN OPINION

(PCT Rule 66)

| | | |
|---|--|---|
| Date of mailing (day/month/year) | | 14.09.2000 |
| Applicant's or agent's file reference 23259.WO/ICB | | REPLY DUE within 3 month(s) from the above date of mailing |
| International application No. PCT/GB99/03580 | International filing date (day/month/year) 29/10/1999 | Priority date (day/month/year) 30/10/1998 |
| International Patent Classification (IPC) or both national classification and IPC A23L1/00 | | |
| Applicant AQUACAL LIMITED et al. | | |


- This written opinion is the **first** drawn up by this International Preliminary Examining Authority.
- This opinion contains indications relating to the following items:
 - ☒ Basis of the opinion
 - ☐ Priority
 - ☒ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
 - ☒ Lack of unity of invention
 - ☒ Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
 - ☐ Certain document cited
 - ☒ Certain defects in the international application
 - ☒ Certain observations on the international application
- The applicant is hereby **invited to reply** to this opinion.

When? See the time limit indicated above. The applicant may, before the expiration of that time limit, request this Authority to grant an extension, see Rule 66.2(d).

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also: For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 bis.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.
- The final date by which the international preliminary examination report must be established according to Rule 69.2 is: **28/02/2001**.

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| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | Authorized officer / Examiner Vernier, F Formalities officer (incl. extension of time limits) Schulz, A Telephone No. +49 89 2399 8112 |
|---|--|



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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

| | | | |
|--|--|---|--|
| Applicant's or agent's file reference 23259.WO/ICB | | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) | |
| International application No. PCT/GB99/03580 | International filing date (day/month/year) 29/10/1999 | Priority date (day/month/year) 30/10/1998 | |
| International Patent Classification (IPC) or national classification and IPC A23L1/00 | | | |
| Applicant AQUACAL LIMITED et al. | | | |

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 5 sheets, including this cover sheet.

- ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

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- VI ☐ Certain documents cited
- VII ☒ Certain defects in the international application
- VIII ☒ Certain observations on the international application

| | |
|---|---|
| Date of submission of the demand 26/05/2000 | Date of completion of this report 13.02.2001 |
| Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 | Authorized officer Vernier, F Telephone No. +49 89 2399 8646  |

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

| | | |
|--|-----------|--|
| (51) International Patent Classification ⁷ : A23L 1/00 | A2 | (11) International Publication Number: WO 00/25602 (43) International Publication Date: 11 May 2000 (11.05.00) |
| (21) International Application Number: PCT/GB99/03580 (22) International Filing Date: 29 October 1999 (29.10.99) (30) Priority Data: 9823885.0 30 October 1998 (30.10.98) GB (71) Applicant (for all designated States except US): AQUACAL LIMITED [IE/IE]; Strand Farm, Currabinny, Carrigaline, Co. Cork (IE). (72) Inventor; and (75) Inventor/Applicant (for US only): ASSOUMANI, Mohamed, Bakri [GB/IE]; Strand Farm, Currabinny, Carrigaline, Co. Cork (IE). (74) Agents: BAILLIE, Iain, C. et al.; Langner Parry, 52-54 High Holborn, London WC1V 6RR (GB). | | (81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>Without international search report and to be republished upon receipt of that report.</i> |
| (54) Title: FOODSTUFF COMPOSITIONS (57) Abstract Residues, preferably purified to avoid content of heavy metals, of corallinaceae are employed in solid or semi-solid foodstuffs to enhance organoleptic and physical properties as well as providing enhanced calcium content. | | |

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

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| BY | Belarus | IS | Iceland | MX | Mexico | US | United States of America |
| CA | Canada | IT | Italy | NE | Niger | UZ | Uzbekistan |
| CF | Central African Republic | JP | Japan | NL | Netherlands | VN | Viet Nam |
| CG | Congo | KE | Kenya | NO | Norway | YU | Yugoslavia |
| CH | Switzerland | KG | Kyrgyzstan | NZ | New Zealand | ZW | Zimbabwe |
| CI | Côte d'Ivoire | KP | Democratic People's Republic of Korea | PL | Poland | | |
| CM | Cameroon | KR | Republic of Korea | PT | Portugal | | |
| CN | China | KZ | Kazakstan | RO | Romania | | |
| CU | Cuba | LC | Saint Lucia | RU | Russian Federation | | |
| CZ | Czech Republic | LI | Liechtenstein | SD | Sudan | | |
| DE | Germany | LK | Sri Lanka | SE | Sweden | | |
| DK | Denmark | LR | Liberia | SG | Singapore | | |
| EE | Estonia | | | | | | |

FOODSTUFF COMPOSITIONS

This invention relates to solid and semi-solid foodstuff compositions particularly foodstuff compositions containing calcium materials.

The fortification of foodstuffs with calcium compounds is well recognised as a means of supplementing calcium in the diet. However, the addition of presently 10 available sources of calcium has been found to result in deterioration in the physical properties of the product to which calcium is added when added in the amounts sufficient to give the desired available calcium so as to ensure an adequate intake of calcium in the diet.

15

In International Patent Application no. PCT/GB98/00142 published as WO/98/33508 there is disclosed the use of a very pure form of corallinaceae for treatment of conditions created by failure of immuno 20 regulation in the body. This has included the use of corallinaceae for the manufacture of a medicament for the treatment of reduced calcium levels and use in manufacturing medicament for raising pH levels in the colon. This application discloses forming emulsions in 25 the manufacture of foods wherein an emulsifier is combined with a residue of a very pure form of corallinaceae (Maërl) and then with an oil phase of a foodstuff which is formed into an emulsion with an aqueous phase. There is reference to the use of these 30 oil products in bakery products. However, this specification relates primarily to inclusion of the residues for nutritive purposes and does not indicate generally the value of this particular material in relation to starch products particularly farinaceous 35 products nor does it discuss improvements in physical, including organoleptic, properties.

Studies of the addition of corallinaceae by-products and residues to foodstuffs have been in relation to nutritive properties (Agro-food-Industry Hi-Tech; September/October, 1997 and see a subsequent article in the September-October 1998 issue). These articles have discussed the properties of calcareous materials in terms of bioavailability of calcium. The high surface area of corallinaceae products appeared to correlate with solubility at various pH's which
10 correlated with calcium absorption and with physiological and biochemical properties arising from such bioavailability. Similarly the later article refers to buffering and similar properties and discusses anti-acid properties, mentions acid uptake in the
15 context of organic juice and particularly structure, texture and mouthfeel in that connection, ie. of beverage.

It has now been found that if a form of
20 corallinaceae is employed in the manufacture of solid and semi-solid foodstuffs much superior results are obtained in the texture of the resulting product. In particular it is possible to add higher amounts of calcium than is possible with other sources of calcium.

25

Without limitation on the invention, it is believed that the advantages of the invention, at least partially, arise from the unusual structure of the calcareous material employed. It is believed that the
30 calcareous material has a porous structure which on hydration or oil absorption collapses to give a very smooth structure. This is analogous to a hydrocolloid or edible gel which holds the fluid phase in extremely small pores. Structure collapse can be achieved with an
35 amount of moisture of 2% by weight.

Other properties of the calcareous material are

film forming properties, adhesiveness and binding properties and non-abrasiveness. These properties are not found in known food grade calcium carbonate materials. These properties were not appreciated from the previous work on the calcareous material based on the invention which was primarily directed to the nutritive and pharmacological properties.

The absorption capacity and binding properties can be of particular advantage in cereal products and may explain the positive effects on stickiness and strength. These effects also assist in applications of carbohydrates (sugars) for example sugar (eg. sucrose or fructose), syrups and honey. There is reduction of water activity and extension of shelf life and improvement of flavour formation by non-enzymatic browning.

The calcareous material does not require prior solubilisation for use in semi-moist or dry products. At about pH 6.5, approximately 19% by weight of the calcium content will ionise and the carbonate portion will slightly increase pH and buffer the system. It is believed this will contribute to homogenous browning (Maillard reactions (with possible Strecker degradations)) during a cooking stage and better flavour formation. These give rise to unexpected advantages in texture, colour, flavour and shelf-life.

The invention therefore has two aspects. The first is the improvement in fatty products where the calcareous material is in the fatty phase and enhances emulsion stability, controls fat crystallisation and enhances organoleptic properties. Incidentally this permits inclusion of sufficient calcareous material to allow incorporation of calcareous material to give in excess of 25% of ERDA requirements.

The second is the improvement as discussed subsequently on non-fatty products of physical and organoleptic properties.

5

This has been found to be of particular application to solid or semi-solid products as distinct from beverages. By a solid or semi-solid product is meant one having significant shape-retaining properties as distinct from flowable liquid compositions which have low or non-existent shape retaining properties so that they would normally be classified as beverages.

While the improvements of the invention can be obtained in high fat compositions, for example emulsified fat products in which the calcareous material is incorporated in an oil phase, improvements are achieved in non-emulsified compositions, for example cheese spreads and yoghurt-type products. Improvement is also found in fatty and non-fatty products of the ice-cream type, ie. intended for consumption while still frozen.

As will be described in detail, a significant improvement is found in farinaceous products when the calcareous materials of the invention are added.

Products of the invention also include confectionery particularly carbohydrate products, ie. products consisting to a significant extent of sugars such as sucrose. These can include candy products, gelatinous products and particularly chocolate based products including cocoa fat products and other fat products and chocolate products such as cocoa. Candy products can be boiled sugar products and other confectionery products. Confectionery products include dessert products including pudding mixes and gelatinous

products. The invention also applies to meat derived products.

A modification of the invention is the use in cosmetic products of the calcareous materials. Such cosmetic products include face masks, scrubs, body wraps and scrubs, ie. those products applied for advantageous effects on the skin as distinct from mere embellishment.

10 The calcareous material used in the invention provides calcium in carbonate form. It contains 34% by weight calcium as compared to 40% by weight for conventional commercial calcium carbonate.

15 By adding the calcareous material according to the invention it is possible to achieve a known RDI (Recommended Dietary Intake) for a specified weight or volume of foodstuff and also, generally, improve the qualities of the product, for example texture, mouth-
20 feel, strength and cooking properties.

A calcareous material useable in the invention is more fully described in the above international patent application. The calcareous material is obtained from
25 corallinaceae.

Corallinaceae, for example Lithothamnium corallioides (Lithothamnium calcareum sometimes known as phymatolithon calcareum), are known seaweeds which are
30 very abundant in certain cold and temperate seas. Once harvested the crude residual product consists primarily of mineral substances, particularly calcium carbonate and magnesium carbonate. The largest component is calcium carbonate, often about 34% by weight. This
35 product is sometimes identified as Maërl though the term Maërl encompasses residues of coralline algae of various members of the order corallinales (Class Rhodophyceae)

including members of corallinaceae for example members of the species *Lithothamnium corallioides*, *Phymatolithon calcareum* and *Lithothamnium glaciale*.

5 Crude corallinaceae (*Lithothamnium corallioides*) residues have been commercially available for use in the prevention of acidosis in intensively fed cows. In French patent FP2 201 040 there is disclosed the use of Maërl which appears to be in the crude form for animal
10 feeds. Such products as have been available until the present time have tended to be relatively impure products frequently from contaminated sources. Usually they contain significant amounts of siliceous materials derived from the original product as dredged and other
15 non-corallinaceae residues for example ground shells of sea-creatures.

The Institute of Oceanography in Paris produced a report on corallinaceae particularly *Lithothamnium* in
20 1989 describing the residual crude product (Maërl) and describing its use in treatment of soil and for animal feed as a dietary supplement and for treatment of water.

Corallinaceae particularly *Lithothamnium*
25 *corallioides* are coralline algae. There are a number of sub-species of corallinaceae particularly *Lithothamnium* differentiated by morphological data but these data can vary depending on local sea bed and weather conditions.

Other known "relatives" include *Phymatolithon calcareum*
30 and in more northerly regions *Lithothamnium glaciale*.

These plants lay down calcium carbonate in their cell walls which gives them a hard stony texture. The living corallinaceae for example *Lithothamnium corallioides* generally show a red colour due to the presence of a
35 pigment phycoerythrin in their structure. When dead the colour is white or yellowish. Corallinaceae for example *Lithothamnium corallioides* occurs naturally in cold and

temperate seas and has been reported in Norway, Canada, Scotland, Ireland and France.

Since compositions of the subject invention are to be used in foodstuffs it is of course important that the corallinaceae which is to be exploited in the invention is derived from a part of the world which does not suffer from heavy pollution. For this purpose corallinaceae particularly *Lithothamnium corallioides* harvested from stocks north of Lonehort Point, Castletownbere, County Cork in the Republic of Ireland have proved very satisfactory but there are also deposits off the West Coast of Galway.

15 Naturally occurring residues of *Lithothamnium corallioides* were harvested at the above site at Lonehort Point, purified and concentrated.

The raw material can be purified by initial extensive washing with sea and fresh water together with removal of extraneous sand, shells, and other debris particularly siliceous debris such as stones. This step usually reduces the material obtained by dredging from the sea bed to about 20% by weight.

25

The cleaned and separated product is then subjected to intensive cleaning by for example, bleaching and sterilising in hydrogen peroxide for from 8 to 24 hours, further washing in water, drying in a sterile fluid bed and final milling under bacterial controlled conditions.

For the purpose of this invention it is important for compositions intended for consumption (edible products) that they comply with Food Regulations, for example in relation to the upper limits for contents of heavy metals. This may result inherently from natural source or from the technique of purification.

The stringent washing conditions can reduce sodium content of the raw product from amounts in the order of well in excess of 1,000 ppm for example amounts up to 5,200 ppm to sodium contents in the low hundreds, for example 300 ppm. Thus there can be a reduction of about 10 fold in the sodium content as compared to raw material.

10 The silica content of this final material is normally not more than 0.5% by weight as compared to a silica content in previously available Maerl from a commercial source, of about 5 % by weight.

15 A representative sample of this purified, concentrate contained the following elements in the following amounts (by weight):

| | | |
|----|-------------|----------|
| | Calcium | 34% |
| 20 | Magnesium | 2.4% |
| | Phosphorous | 0.08% |
| | Potassium | 0.10% |
| | Sulphur | 0.45% |
| | Iron | 25 ppm |
| 25 | Boron | 16.5 ppm |
| | Fluorine | 200 ppm |
| | Sodium | 310 ppm |
| | Manganese | 125 ppm |
| | Nickel | 30 ppm |
| 30 | Cobalt | 6 ppm |
| | Copper | 10 ppm |
| | Lead | 460 ppb |
| | Zinc | 37 ppm |
| | Selenium | 1 ppm |
| 35 | Molybdenum | 39 ppm |
| | Iodine | 160 ppm |
| | Arsenic | <1 ppm |
| | Chromium | 13 ppm |
| | Cadmium | 0.2 ppm |
| 40 | Mercury | <50 ppb |
| | Aluminium | <5 ppm |

According to the present invention there is provided use in a solid or semi-solid foodstuff of a

material to improve organoleptic and physical properties and calcium content characterised in that the material is a calcareous residue of corallinaceae with a content of heavy metals below the upper limits acceptable for edible products.

The calcareous residue can be incorporated in a foodstuff having an emulsified oil or fat phase into which is incorporated the corallinaceae residue and 10 which has improved organoleptic properties as compared to the same product free of said residue but advantages also exist for a foodstuff containing fat material in which the corallinaceae residue is distributed generally in the foodstuff.

15

Particularly valuable foodstuffs in which the invention has advantage is ones to be consumed in frozen form. The invention is also applicable to yoghurt products.

20

The invention is also particularly applicable to carbohydrate products including desserts, confectionery and similar products or chocolate products.

25 In non-fat products the calcareous material is preferably added by a carbohydrate (sugar) water phase.

A modification of the invention is use of the calcareous material in cosmetic products, which products 30 have advantages on the skin as distinct from mere decorative effects.

The foodstuff can contain a sufficient proportion of the calcareous material as defined above derived from 35 corallinaceae to provide a substantial proportion of the Recommended Dietary Intake of calcium in the daily diet.

The foodstuff in question is primarily intended for

human beings although the invention could be applied to foodstuffs for animals.

Particular foodstuffs are starch based foodstuffs, especially those derived from farinaceous materials i.e. those based primarily on wheat or similar farinaceous grains. Particular products in which the calcareous material can be employed include bread, so-called biscuits or wafers, the various forms of pasta including noodles, breakfast cereals and extruded farinaceous products and so-called snack foods.

Particularly in relation to pasta and as discussed in an article by J Smewing on the Texture of Pasta in Cereal Foods World January 1997 volume 42 no. 1 pages 8 through 12 microstructure changes profoundly affect the properties of the resulting pasta and changes in the components can radically change the hydration characteristics. In that article there are described assessment of various product characteristics both cooked and uncooked pasta products.

The proportion of calcareous product added can depend on the final desired calcium Recommended Dietary Intake or the improvement in physical (eg) organoleptic properties but for example can range up to 4 or 5% by weight of the basic raw materials in forming the final food product. The preferred range is 0.5 to 3% by weight most preferably 1 to 2% by weight more particularly it is up to about 1.6% by weight of the product. For example in biscuits intended to supplement a diet with calcium one can employ approximately 20 grams per so-called biscuit representing about 2% of the final product.

35

The addition of the calcareous product used in the invention as compared to the results when other sources

of calcium are used not only improved the properties of baked products but, in some instances, has also been found to provide a buffering effect in the stomach and also appears to provide anticariogenic effects. It is believed that this may partly arise from protection against acid in the mouth.

The invention will now be illustrated by the following examples which are not however intended to limit the scope of the invention. The Calcium Product (calcareous product derived from corallinaceae) employed is a commercial product prepared from Lithothamnium corallioides residues as described above and having the analysis set out above and identified commercially as AquaMin. The coated Calcium Product is the calcareous product coated with a mono-diglyceride.

Example 1

Fortification of pasta with calcium.

20

A conventional pasta product of the spaghetti nature manufactured from durum or other hard wheat flour was employed.

25 Manufacture of Pasta:

A basic recipe for pasta was used.

Durham Wheat Semolina (766g) + Tap Water (234g) → 1Kg
30 of Pasta.

Mix for 10 min in mixing chamber of the pasta press



Rest for 5 min



35

Warm up die : Extrude

12



Cook for four min



Cool for 30-60 sec under running tap water

5



Analyse

The following batches of conventional pasta were made and analysed.

1. Control (no added Calcium).
2. Pasta + 0.73% Calcareous Product (40% R.D.I./150g serving).
- 15 3. Pasta - 1.26% Calcareous Product (70% R.D.I./150g serving).
4. Pasta - 1.4% Coated Calcareous Product (70% R.D.I./150g serving).
5. Pasta + 1.07% Calcium Carbonate control (70% R.D.I./150g serving).
- 20

[R.D.I. - Recommended Dietary Intake]

100g of pasta was then cooked in 500ml of water for four
25 minutes, the pasta was then analysed for firmness and stickiness using a texture analyser (AACC 16-50 standard method).

Results:

30

| Sample | Firmness (Force g) | Stickiness (Force g) |
|-----------|--------------------|----------------------|
| Batch one | 333.022 | -1019.63 |
| Batch two | 377.902 | -1019.927 |

| | | |
|-------------|---------|-----------|
| Batch three | 300.144 | -1018.654 |
| Batch four | 310.046 | -1018.367 |
| Batch five | 291.144 | -1017.703 |

The control batch and batch two (40% R.D.I.) were made and analysed on the same day. The test results 5 showed that addition of Calcareous Product increased the firmness of the pasta and reduced the stickiness when compared to the control.

At a higher level of Calcareous Product addition, 10 additional water was added (5ml/1 Kg pasta) to prevent the pasta becoming too firm. Therefore a direct comparison cannot be made between batches 1,2 and 3,4,5.

Batch five was significantly stickier than any of 15 the other batches of pasta. This was evident in handling the pasta as strands tended to stick together. This did not happen to the other batches.

The organoleptic qualities - colour, volume, 20 speckledness, glossiness and bulkiness - of each batch of pasta were similar and it was impossible to detect any differences in taste between the batches.

The fortification of fresh pasta with a Calcareous 25 Product as employed in this invention was very successful, increasing the strength of the pasta and reducing the stickiness.

Example 2

30 Fortification of biscuits with Calcium

Four batches of biscuits were made using the

following recipe:

| | |
|--------|---------------------------------|
| 400g | Confectionery Flour |
| 166.8g | Fat |
| 5 140g | Sugar |
| 20g | Syrup |
| 2.8g | Salt |
| 2.0g | Ammonium Bicarbonate |
| 2.0g | SSL (Sodium Stearoyl Lactylate) |
| 10 73g | Water |

1. Batch one: Control - no added Calcium.
2. 1.8% Calcareous Product.
3. 2.0% Coated Calcareous Product } 40% Calcium
15 R.D.I.
- per serving
4. Calcium Carbonate Control.

* one serving of biscuits is three biscuits (20g in 20 weight each).

The biscuits were cooked for exactly eleven minutes and then analysed.

25 The following parameters were examined: friability, water activity (Aw) and colour.

| Sample | Friability | Aw |
|-------------|------------|-------|
| Batch one | 3483.41 | 0.306 |
| Batch two | 4275.13 | 0.353 |
| Batch three | 3406.77 | 0.335 |
| Batch four | 1333.66 | 0.520 |

Colour was measured using LAB values.

- L. Brightness
5 A. Red
B. Yellow

| Sample | L value | A value | B value |
|-------------|---------|---------|---------|
| Batch one | 60.64 | 10.52 | 33.78 |
| Batch two | 63.66 | 9.42 | 31.77 |
| Batch three | 63.63 | 9.60 | 31.83 |
| Batch four | 72.87 | 3.33 | 33.18 |

10 Friability

The results showed that addition of Calcareous Product increased the friability of the biscuit when compared with the control (3483.4-4275.1) and addition of coated Calcareous Product decreased the friability of the
15 biscuit. However these differences could not be detected by a taste panel. The friability of the biscuits fortified with Calcium Carbonate were significantly reduced and this was very obvious to the taste panel who felt the biscuits tasted soft/gone off.

20

Water Activity

The water activity of the biscuits fortified with Calcium Carbonate was significantly increased when compared with the control. Addition of either
25 Calcareous Product or coated Calcareous Product did not have a significant effect on the water activity of the biscuits.

Colour

The LAB values of the biscuits were measured using a Minolta colour meter.

The lightness of the biscuits fortified with Calcareous Product and coated Calcareous Product were marginally increased, whereas the biscuits fortified with Calcium carbonate increased from 60.64-72.87.

The red colour of the biscuits fortified with Calcium Carbonate was significantly reduced when compared with the control, Calcareous Product and coated Calcareous Product had little effect on this parameter.

The yellow colour of the biscuits was marginally reduced in both the biscuits with additional Calcareous Product, Calcium carbonate did not effect this value.

The taste panel were in agreement that there was very little difference in the appearance and taste of the biscuits fortified with Calcareous Product and coated Calcareous Product when compared with the control. Most people were unable to identify which biscuits had the additional Calcium. However the biscuits fortified with Calcium Carbonate were pale in colour, soft and unpalatable to taste (loss of sweet flavour).

EXAMPLE 3**CALCIUM FORTIFICATION OF SPREADS**

Margarine

Vegetable Oil
Spread**Fat Content**

80% minimum

70-20%

Nature of Fats

Saturated

Unsaturated



High Melting Point

Low Melting Point

Emulsion

There are two phases :

Water Phase

Oil Phase

The two phases are mixed to build the emulsion. Emulsion building requires energy input in the form of mechanical agitation, ultrasonic vibration or heat.

1. Emulsion Building (3 to 5 minutes)



2. Quick Chilling To 15°C



3. Fat Crystallisation

Method

AquaMin must be added to the oil phase : the oil will go inside the pores. This will help stabilise the emulsion.

The order of mixing is critical for the addition of AquaMin to this type of emulsion structure spread.

If AquaMin is added to the water phase first, then the water enters the porous structure and these pores become polar. The outside surface of AquaMin is also polar, so that when this is now mixed with the oil, which is hydrophobic, this will destabilise the emulsion.

If however, AquaMin is added to the oil phase first, then the oil enters the pores and due to the oil viscosity it is retained inside. The oil, being hydrophobic, now makes the internal pores hydrophobic. The outside AquaMin surface area is still polar, so now when added to the water phase, which is also polar, a stable emulsion will result.

After the emulsion building stage, chilling and fat crystallisation follow and during the crystallisation stage, AquaMin promotes the formation of the β' crystal form. This crystal structure is most desirable, as it requires less energy to melt than the larger β form and is more stable than the smaller lower energy α form and consequently, the β' crystals give the spread a better mouth feel. As a result of this, in the spread AquaMin has excellent uniform calcium distribution, with no detection of the presence of particles in the mouth.

The only technical issues to be aware of in terms of the impact on the quality of the finished spread are:

- AquaMin's high buffering capacity may affect the titratable acidity of the spread, so in this case it will be necessary to monitor the titratable acidity during the process and compensate through the addition of lactic acid.
- At addition rates above 2%, the off-white colour of AquaMin may affect the colour of the spread, so here it will be necessary to add beta-carotene to the formulation to counter this.

Both issues are dependent on the level of AquaMin addition and will vary on the composition of the spread in terms of fat content, but are easily overcome using ingredients that are universally used during the production process.

EXAMPLE 4**CALCIUM FORTIFICATION OF
CHEESE SPREADS**

A cheese spread was made using a standard recipe of :

Young, Medium-Ripe and Over-Ripe Cheddar Cheese

Water

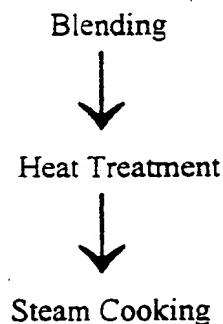
Butter

Whey Powder

Emulsifier

Salt

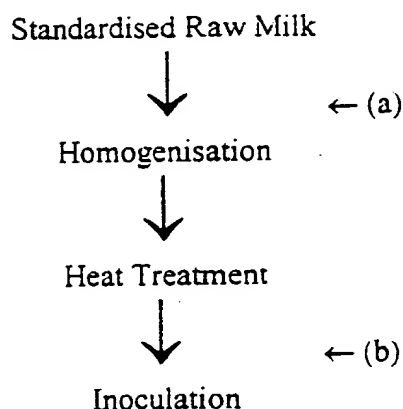
Preservatives

Method

AquaMin is added during the cooking stage and disperses homogeneously throughout the spread. AquaMin can be easily incorporated at levels of 1-2% without any adverse effects. At 2% AquaMin addition in a 200g tub, a 15g serving will provide 12.75% of the RDI for Calcium (the European Union RDI for Calcium is 800mg/day).

EXAMPLE 5**CALCIUM FORTIFICATION OF YOGHURT**

In the case of yoghurt production, industrial production typically follows the following process :

**Method**

AquaMin can be added either (a) before homogenisation or (b) after heat treatment. (a) is preferred as calcium ionisation will be improved and will promote the Ca^{++} interaction with denatured α s-casein. This can result in a slight increase in viscosity.

In the case of stirred yoghurt with fruit, AquaMin can be added to the fruit purees before heat treatment. Ca^{++} will help stabilise fruit puree through the formation of calcium pectate.

This aspect of the invention relates to solid or semi-solid yoghurt compositions as distinct from beverages based on yoghurt.

EXAMPLE 6**CALCIUM FORTIFICATION OF ICE-CREAM**

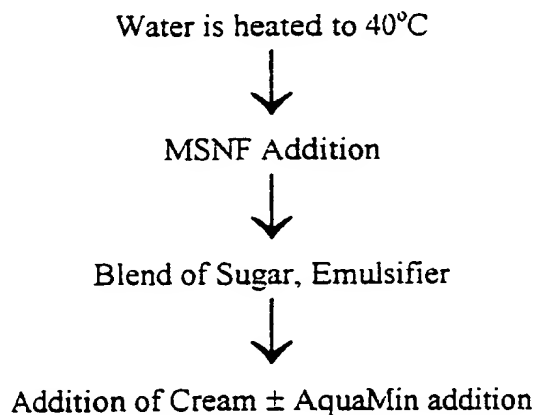
Ice-cream was made using a standard recipe as follows :

| | |
|------------|------|
| Fat | 17% |
| MSNF | 11% |
| Sugar | 14% |
| Emulsifier | 0.5% |
| Water | 57% |

Two batches of ice-cream were made :

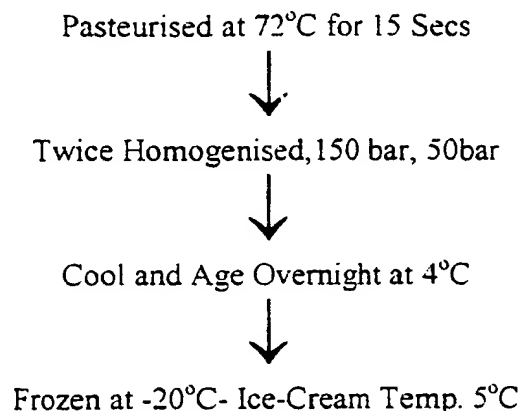
1. Control
2. + 0.79% AquaMin

(Addition of AquaMin provides 100% Calcium fortification in a 200g serving)

Method

/.....

The ice-cream solution is mixed continuously using a Silverson mixer. The solution is then :



During freezing samples of ice-cream were taken from the different batches at different times to ensure homogenous dispersion.

The following parameters of the ice-cream were examined

1. Calcium Analysis
2. Sensory Analysis
3. Colour
4. Over-Run

1. Calcium analysis confirmed that the calcium was homogeneously dispersed throughout the ice-cream.

| Sample | Observed (PPM) |
|-------------------|----------------|
| Control | 1670 |
| AquaMin | 4070 |
| Calcium Carbonate | 3950 |

Samples of ice-cream taken at different stages during the production had similar calcium levels

2. A sensory analysis of the ice-cream was carried out in a local university under controlled conditions in their sensory analysis unit (report available upon request). Panellists were asked to assess ice-cream using the following parameters :

Sample :

Taste (1 = very poor, 5 = very good)

Grittiness (1 = very gritty, 5 = not gritty)

Overall acceptability (1 = worst, 5 = best)

/.....

19 panellists took part in this analysis and the results were as follows

| Sample | Taste | Grittiness | Acceptability | Preference |
|-----------------------------|-------|------------|---------------|------------|
| A, AquaMin | 3.2 | 4.6 | 3.6 | 15 |
| B, Calcium Carbonate | 2.8 | 4.3 | 2.9 | 1 |
| C, Control | 2.7 | 2.9 | 2.3 | 3 |

From the results it is clear that AquaMin fortified ice-cream is predominant - 79% of panellists preferred the ice-cream fortified with AquaMin. The control and the ice-cream fortified with calcium carbonate lagged behind, with only 16% and 5% of preferences respectively. The ice-cream fortified with AquaMin scored highest on all parameters of taste, grittiness and acceptability.

3. Colour was measured using a Minolta colour meter and the results were expressed using LAB values :

L = Lightness
A = Red Colour
B = Yellow Colour

| Sample | L value | A value | B value |
|--|---------|---------|---------|
| Control | 94.03 | -3.21 | 11.82 |
| AquaMin fortified ice-cream | 94.39 | -2.87 | 11.37 |
| Calcium carbonate fortified ice-cream | 95.61 | -2.84 | 10.83 |

A statistical Student's t- Test was carried out on these values (9 values for each sample) and the results of the test showed that there was a significant difference between the control and the ice-cream fortified with calcium carbonate for each of the three parameters. AquaMin only had a significant effect on the A value of the ice-cream, it did not effect the L or B values.

4. A further batch of ice-cream was made to assess if calcium addition effected the over-run properties of the ice-cream. Production conditions were kept constant and it appears that addition of calcium did not have a significant effect on the over-run properties. The control, AquaMin fortified ice-cream and the calcium carbonate fortified ice-cream had the following over-run of 130%, 139% and 136% respectively.

EXAMPLE 7**CALCIUM FORTIFICATION OF LOW FAT
ICE-CREAM**

Low fat ice-cream was made using a standard recipe.

Three batches of ice-cream were made :

1. + 0.8% AquaMin
2. + 0.6% Calcium carbonate
3. Control

(Addition of AquaMin provides 100% Calcium fortification in a 200g serving)

Processing conditions were kept constant and it was determined that there were no differences in the over-run between the different batches.

A sensory analysis of the ice-cream was carried out under controlled conditions (report available upon request) during which panellists were asked to assess samples from the three batches ice-cream using the following parameters :

Sample :

Sweetness (1 = not sweet, 5 = extremely sweet)

Creaminess (1 = not creamy, 5 = extremely creamy)

Iciness/Coarseness (1 = very icy, 5 = not icy)

Overall acceptability (1 = worst, 5 = best)

17 panellists took part in this analysis and the results were as follows :

| | Sample A +0.8% AquaMin | Sample B +0.6% CaCO ₃ | Sample C Control |
|--------------------------|------------------------------|--|---------------------|
| Sweetness | 3.29 | 3.12 | 3.18 |
| Creaminess | 4 | 3.35 | 3.53 |
| Iciness/Coarseness | 4.71 | 4.24 | 4.41 |
| Overall Acceptability | 3.18 | 3.29 | 3.47 |
| Preference | 7 | 3 | 7 |

/.....

A statistical Student's t- Test was carried out on the above data and this showed that there was not a significant difference in the results between sweetness and overall acceptability of the product. However differences were evident in the parameters of creaminess and iciness/coarseness. Sample A was significantly creamier and significantly less icy/coarse than Samples B and C.

EXAMPLE 8**CALCIUM FORTIFICATION OF SWEETS**

Handmade sweets were made using a traditional recipe of :

Sugar
Water
Cream of Tartar
Flavour
Malic Acid
Food Colours

AquaMin was added to this recipe at a level of 3.5%.

The sugar and water is boiled, allowed to cool, and as it solidifies on a metal bench, the AquaMin, Malic Acid, flavour and dyes are splashed on and folded into the mixture.

It is necessary to add extra Malic Acid (AquaMin: Malic Acid, 3:1) to counteract a bland flavour.

A variety of flavours and colours were used.

Each sweet weighs approximately 3.5g and contains 40 mgs of Calcium.

EXAMPLE 9

COSMETICS

The unique properties of AquaMin make it extremely suitable for a wide range of cosmetic applications, where it can be incorporated into face masks and scrubs and body masks, wraps and scrubs. The high level of naturally occurring trace elements present in AquaMin can regenerate and mineralise the epidermis.

The key properties of AquaMin relevant for cosmetics are :

1. Mineral Content – AquaMin contains a wide range of natural minerals including Calcium, Magnesium, Iodine, Sodium, Boron, Phosphorous, Sulphur, Iron, Sodium, Nickel, Cobalt, Zinc etc. These can revitalise skin to leave as well as playing an important role in our physiology : Calcium strengthens teeth, bones, fingernails and balances the water level of tissue. Magnesium helps combat stress and relaxes muscle contractions. Iodine and Sodium assist in regulating our metabolism. The combination of Calcium, Magnesium and Boron present in AquaMin can alleviate aches and pains in joints when used as a therapeutic seaweed wrap.
2. Particle Size & Structure – AquaMin has an average particle size of 2.5-5 microns, which makes it ideal for cosmetic formulations requiring fine particulate size such as in make-up foundations and sun blocks. Once hydrated, AquaMin's structure collapses to give an extremely smooth texture, close to that of talcum powder.
3. Oil Absorption – AquaMin's high surface area enables it to readily absorb essential oils, up to a level of 40%, thereby cleansing the skin. Similarly, AquaMin can be combined with herbal essences and extracts due to it's absorption property.

Face Masks

A typical formulation is as follows :

| | |
|---------------|--------------------------------------|
| AquaMin F | 55-60% |
| Water | 40-45% |
| Essential Oil | 3-4 drops (Sandalwood, Teatree etc.) |

The above ingredients are mixed to form a paste, which is applied directly to the facial skin and allowed to dry for 5-10 minutes, after which it is washed off with warm water. After removal, the skin is cleansed, smooth and soft to touch, the effects of which can last for several days. /.....

Body Wraps

Typically 250g is required for an average body wrap. In some cases, AquaMin can be combined with other seaweed products such as laminaria and/or fucus.

1. Weight Loss :

The body is wrapped tightly in bandages which have been soaked in an AquaMin paste. These are left in place for 45 minutes and then removed. The effect of the body wrap can reduce 1-2 inches in overall skin measurements and at the same time the skin is very soft.

2. Revitalisation :

The body is covered in an AquaMin paste and then wrapped in plastic and a heated blanket for 40 minutes. Application takes place in a relaxing environment with pleasant music and soft lighting. Afterwards, the paste is then washed off and the skin is left smooth, soft and replenished.

3. Inflammation Therapy :

The AquaMin paste is applied directly to a specific joint which may be swollen, arthritic or bruised. After massaging in lightly, the wrap is left for some time prior to removal. The result is an observed reduction in any swelling and associated aches and pains.

CLAIMS:

1. Use in a solid or semi-solid foodstuff of a material to improve organoleptic and physical properties and calcium content characterised in that the material is a calcareous residue of corallinaceae with a content of heavy metals below the upper limits acceptable for edible products..

2. A use according to claim 1 wherein the product is a foodstuff having an emulsified oil or fat phase into which is incorporated the corallinaceae residue and which has improved organoleptic properties as compared to the same product free of said residue.

3. A use according to claim 1 in which the foodstuff containing fat material in which the corallinaceae residue is distributed generally in the foodstuff.

4. A use according to claim 1 in which the foodstuff is a foodstuff to be consumed in a frozen form.

5. A use according to claim 1 in which the foodstuff is a carbohydrate product.

6. A use according to claim 3 in which the foodstuff is a chocolate product.

7. A use according to claim 5 in which the foodstuff is a farinaceous product.

8. A use according to claim 7 in which the foodstuff is primarily composed of a starch based material.

9. A cosmetic material with a content of a residue of corallinaceae to enhance the properties on skin.